

1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 5

LANDFILL LEACH SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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G. C. RONAN, DIRECTOR  
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Ministry of the Environment

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ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 5

LANDFILL LEACH SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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and J C HIPFNER (editors)

Inorganic Trace Contaminants Section  
Laboratory Services Branch  
Ministry of the Environment

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## INORGANIC TRACE CONTAMINANTS SECTION

## SUMMARY

**I. Introduction**

The Inorganic Trace Contaminants Section of the Ministry of the Environment, Laboratory Services Branch is responsible for the analysis of a wide variety of sample types for metals and non-metals. The use of sensitive instrumentation and methodologies appropriate to the sample matrix, combined with quality assurance programs, ensures that the Section is able to maintain a high standard of analytical performance. This performance is monitored through regular internal quality control and assurance programs as well as participation in interlaboratory round-robbins. This QA report summarizes the methodologies used for analysis of these samples and the supporting internal quality assurance data.

This report is assembled in sections that reflect the analyses performed on different sample matrices in support of the programs of the Ministry of the Environment. Coincidentally, these divisions also reflect the supervisory responsibilities within the Section.

**II. Quality Control and Assurance**

The objectives of the quality control and assurance programs are to ensure that all of the components of the analytical process are under control and to ensure immediate detection and correction of unacceptable analytical performance. The program monitors all of the reagents, instrumentation, calibration and recovery components of the analytical system.

**A. Quality Control**

Quality control of the analytical process takes place at the instrument level and is intended to ensure that the instrumentation is operating according to established criteria. This control function ensures that instrument calibration, standardization, slope and intercept, and instrumental drift meet these criteria.

**B. Quality Assurance**

Quality assurance of the analytical process takes place after the results have been generated and is intended to ensure that the analytical protocols of sample preparation and digestion have been carried out correctly. This control function ensures that reagent blanks, digested standards, sample duplicates and recovery materials meet established response criteria.

### III. Report Format

The report consists of one page method summaries and one page data summaries of blanks, between-run controls and within-run duplicates in formats that are common to all of the parameter/matrix combinations. The method summaries give a brief outline of the sample preparation and measurement procedures. The data summaries consist of annual mean values with standard deviations.

For the within-run duplicates, the data set is subdivided into ranges approximating 0 to 20 %, 20 to 50 % and 50 to 100% of the analytical range. All results for duplicates reported to the data base that are "<" or that have been diluted into the range are excluded from the statistical analysis.

The standard deviations for blanks and between-run controls are calculated using formula I. Formula II is used for the calculations for within-run duplicates.

$$sd = \sqrt{[(\sum x^2 - (\sum x)^2)/n]/(n-1)} \dots\dots I$$
$$sd = \sqrt{\sum d^2/2n} \dots\dots II$$

where : x = the individual values; n = the number of events  
d = the differences between pairs of duplicates

The data is stored in a personal computer using BMB Manager II files. All data manipulations, reports generated etc, are performed using applications written in Manager Math.

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## 5. Landfill

### 5.1 Landfill Leach Samples

Landfill samples are samples collected in bore holes and surface runoff in and around landfill sites in order to monitor the materials that may be leaching from the fill site. In contrast to soil leachate samples, these samples are a result of natural leaching of landfill and other material.

Table 5.1 summarizes the parameters determined, the preparation methods used and the instrument types used for the analysis of landfill samples.

TABLE 5.1

Parameter	Collection Device	Preparation	Analysis
Metals	Plastic or glass jars	Acid digest	ICP - AES
Anions	Plastic or glass jars	Filtration	IC
Cyanide	Plastic or glass jars	Distillation	Colorimetry
Sulfide	Plastic or glass jars		Colorimetry
Mercury	Plastic or glass jars	Acid digest	Cold vapour AAS
Hydride	Plastic or glass jars	Acid digest	Hydride AAS

### 5.2 Landfill Quality Assurance

Sub aliquots of landfill samples are analysed separately to generate duplicate results. Blanks consist of the digestion acid or distilled water as appropriate.

Landfill samples frequently have solid, aqueous and organic liquid phases. In general only the aqueous phase is sampled for inorganic analyses, and the QA materials reflect this position. Composite QA samples are normally spiked to achieve measureable levels of all components determined.

Table 5.2 summarizes the QA materials used when landfill samples are being analysed.

TABLE 5.2

Sample Designation	Type	Parameter
qcal,qca2	Standard solution	Cyanide
qcd	Standard solution (0.2 mg/L)	Cyanide
QCLF-3	composite landfill (prep 1985)	ICP Metals
475-3	EPA solution	Hydride Metals

**ANALYTICAL PROCEDURE**  
**Inorganic Trace Contaminants Section**

TEST NAME:Aluminum TEST CODE:ALUT SAMPLE TYPE:Landfill Lch.  
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE: 533AA0  
REVISION NO: Original DATE:  
NATURE OF LAST REVISION:

## SAMPLE HANDLING:

Quantity Required- 500 ml  
Container- Glass or plastic container  
Preservative- do not preserve  
Other- Field filtered samples preferred

**SAMPLE PREPARATION:**Partial Extn.- Total Extn.-Yes % Extracted-100%

Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

**INTERFERENCES:** Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 3 places after decimal

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 50 mg/L

#### **Resolution:**

#### Sensitivity:

Instrument Detection Limit: 0.08 mg/L

#### **PERFORMANCE CHARACTERISTICS:**

Routine Operating Range = 0.03 - 5.0 mg/l.

Accuracy- Not known: no reference standards available

#### Precision of Controls-

**A** 3.30 mg/l

mean 2.30 mg/L  
std. dev. 0.63 mg/L

std. dev. 0.62 mg/L  
R S D 27.3 %

R.S.D. 27.2 % Precision of Duplicates-low range mid range high

icates low range Mid range high ra  
s.d. 0.035 0.783 0.226

s.d. 0.035 0.793  
mean 0.351 1.518

W 0.05 mg/l mean 0.251 T 0.35 mg/l

CONTROL LIMITS: Analysis repeated if values for in-house reference  
OCLE1 exceed  $\pm 15\%$  PSD

**REMARKS.**

- REMARKS:

  - % extracted - assumed 100% as material originally in solution.
  - Detection Limit - 3x std. dev. of low range within-run duplicates.
  - Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM IN LANDFILL

Operating Range = 0.030 to 5.0 mg/L

IN - RUN DUPLICATES

---

Range	<0.030	0.030 to 1.00	1.00 to 2.50	2.50 to 5.0	>5.0
no.	34	86	10	3	4
s.w.		0.0348	0.7929	0.0357	
mean		0.2511	1.5494	3.5769	

---

QA CONTROL SAMPLES

---

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	204	2.301	0.6249	27.15

---

BLANKS

---

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	116	0.108	0.0711

---

ANALYTICAL PROCEDURE  
Inorganic Trace Contaminants Section

TEST NAME: ARSENIC  
UNIT: Biomaterials

TEST CODE: ASUT      SAMPLE TYPE: Landfill Lch.  
SUPERVISOR: R. Sadana

METHOD CODE: 510BF3

REVISION NO:

NATURE OF LAST REVISION:

DATE: January, 1983

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Glass bottle with bakelite screw cap (16 oz.)

Preservative- 1ml conc. HNO<sub>3</sub> for sample filling 16 oz. bottle

Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-yes % Extracted-

Procedure- Digest samples by mixing 20 ml of sample and 4 ml of acid 6:3:1 (HNO<sub>3</sub>:HClO<sub>4</sub>:H<sub>2</sub>SO<sub>4</sub>) and heating on a hot plate until dense white fumes are formed. Allow sample to cool and add .5 ml of H<sub>2</sub>O and 2.5 ml HCl. Transfer the contents of the beaker to a test tube graduated at the 20 ml mark and bring to volume with washing. Shake and analyze by automated hydride-F.A.A.S. technique.

INTERFERENCES: Excessive concentrations of Cu, Fe, Ni

REPORTING RESULTS: mg/L-2 dec. if <10, 1 dec. if 10-100, 0 dec. if >100

INSTRUMENTATION: Atomic Absorption Spectrophotometer (Varian 1200) with strip chart recorder, peristaltic pump, auto-sampler, open-ended and heated quartz 'T' cell (0.6x10 cm), and gas-liquid separator.

Calibration Range: 0 to 40 ng/ml

Resolution: 0.01 absorbance

Sensitivity: 20 ng/ml gives 0.150 Abs.

Instrument Detection Limit: 1 ng/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.001 to 0.04 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.412 mg/L	
std. dev.	.015 mg/L	
R.S.D.	3.7 %	

Precision of Duplicates-low range      mid range      high range

s.d.	0.0002	0.0007	
mean	0.002	0.009	0.669

W .001 mg/L

T .005 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ARSENIC IN LANDFILL

Operating Range = 0.001 to 0.04 mg/L

IN - RUN DUPLICATES

---

Range	<0.001	0.001 to 0.01	0.01 to 0.02	0.02 to 0.04	>0.04
no.	18	19	1	1	2
s.w.		0.0002	0.0007	0.0000	
mean		0.0020	0.0090	0.0320	

---

QA CONTROL SAMPLES

---

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
475-3	41	0.412	0.0154	3.74

---

BLANKS

---

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

---

ANALYTICAL PROCEDURE  
Inorganic Trace Contaminants Section

TEST NAME:Barium                    TEST CODE:BAUT                    SAMPLE TYPE:Landfill Lch.  
UNIT:Ind.,Dom.,Landfill Wastes                    SUPERVISOR:J. Pimenta

METHOD CODE: 533AAO  
REVISION NO: Original                    DATE:  
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 500 ml  
Container- Glass or plastic container  
Preservative- do not preserve  
Other- Field filtered samples preferred

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-100%  
Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS:mg/L to 3 places after decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.008 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.005 - 2.0 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

	A	B
mean	.215 mg/L	
std. dev.	.027 mg/L	
R.S.D.	12.5 %	
Precision of Duplicates-low range	mid range	high range
s.d.	.007	.015
mean	.085	.670
W 0.01 mg/L	T 0.05 mg/L	1.004
		1.106

CONTROL LIMITS: Analysis repeated if values for in-house reference QCLF1 exceed  $\pm 15\%$  RSD.

REMARKS:

- Extraction efficiency assumed to be 100% as material originally in solution. No ref. stds. available.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

BARIUM IN LANDFILL

Operating Range = 0.005 to 2.0 mg/L

IN - RUN DUPLICATES

---

Range	<0.005	0.005 to 0.40	0.40 to 1.00	1.00 to 2.0	>2.0
no.	11	114	8	2	2
s.w.		0.0074	0.0145	1.0035	
mean		0.0852	0.6696	1.1060	

---

QA CONTROL SAMPLES

---

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	204	0.215	0.0268	12.50

---

BLANKS

---

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	84	0.005	0.0116

---

**ANALYTICAL PROCEDURE**  
**Inorganic Trace Contaminants Section**

TEST NAME:Cadmium            TEST CODE:CDUT            SAMPLE TYPE:Landfill Lch.  
UNIT:Ind.,Dom.,Landfill Wastes            SUPERVISOR:J. Pimenta

METHOD CODE: 533AAO

REVISION NO: Original

DATE:

NATURE OF LAST REVISION:

**SAMPLE HANDLING:**

Quantity Required- 500 ml

Container- Glass or plastic container

Preservative- do not preserve

Other- Field filtered samples preferred

**SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-90%**

Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

**INTERFERENCES:** Several, compensated for by the computer program.

**REPORTING RESULTS:**mg/L to 3 places after decimal.

**INSTRUMENTATION:** Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.005 mg/L

**PERFORMANCE CHARACTERISTICS:**

Routine Operating Range- 0.002 - 0.1 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

A

B

mean 1.760mg/L

std. dev. 0.129mg/L

R.S.D. 7.3 %

Precision of Duplicates-low range mid range high range

s.d. 0.0012 0.0003

mean 0.0068 0.0917

W .002 mg/L T .010 mg/L

**CONTROL LIMITS:** Analysis repeated if values for in-house reference QCLF1 exceed ±15% RSD.

**REMARKS:**

- % extracted - based on spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.

- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN LANDFILL

Operating Range = 0.002 to 0.1 mg/L

IN - RUN DUPLICATES

---

Range	<0.002	0.002 to 0.02	0.02 to 0.05	0.05 to 0.1	>0.1
no.	125	9	0	1	2
s.w.		0.0012	0.0000	0.0003	
mean		0.0068	0.0000	0.0917	

---

QA CONTROL SAMPLES

---

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
OCLF-3	204	1.760	0.1285	7.30

---

BLANKS

---

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	43	0.003	0.0036

---

**ANALYTICAL PROCEDURE**  
Inorganic Trace Contaminants Section

TEST NAME: Calcium            TEST CODE: CAUT            SAMPLE TYPE: Landfill Lch.  
UNIT: Ind., Dom., Landfill Wastes            SUPERVISOR: J. Pimenta

METHOD CODE: 533AA0

REVISION NO: Original

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 500 ml

Container- Glass or plastic container

Preservative- do not preserve

Other- Field filtered samples preferred

SAMPLE PREPARATION: Partial Extn.-      Total Extn.-Yes % Extracted-100%

Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 3 places after decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 50 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.02 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.2 - 100 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

	A	B
mean	304.1 mg/L	
std. dev.	34.2 mg/L	
R.S.D.	11.3 %	

Precision of Duplicates-low range      mid range      high range

s.d.	0.4	0.6	2.2
mean	8.6	34.9	73.0

W 1.0 mg/L                                  T 5.0 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference QCLF1 exceed  $\pm 15\%$  RSD.

REMARKS:

- % extracted - assumed 100% as material in solution.

- Detection Limit - 3x std. dev. of low range within-run duplicates.

- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM IN LANDFILL

Operating Range = 0.200 to 100.0 mg/L

IN - RUN DUPLICATES

Range	<0.200	0.200 to 20.00	20.00 to 50.00	50.00 to 100.0	>100.0
no.	3	14	16	45	59
s.w.		0.4014	0.5734	2.1665	
mean		8.5883	34.9423	72.9586	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
OCLF-3	202	304.083	34.2314	11.26

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	43	0.279	0.3469

ANALYTICAL PROCEDURE  
Inorganic Trace Contaminants Section

TEST NAME: Chromium TEST CODE: CRUT SAMPLE TYPE: Landfill Lch.  
UNIT: Ind., Dom., Landfill Wastes SUPERVISOR: J. Pimenta

METHOD CODE: 533AA0  
REVISION NO: Original DATE:  
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 500 ml  
Container- Glass or plastic container  
Preservative- do not preserve  
Other- Field filtered samples preferred

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-93%  
Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 3 places after decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.01 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 - 1.0 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

	A	B
mean	1.73 mg/L	
std. dev.	0.15 mg/L	
R.S.D.	8.5 %	

Precision of Duplicates-low range mid range high range

s.d.	0.012	0.025
mean	0.036	0.308

W 0.02 mg/L T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference QCLF1 exceed  $\pm 15\%$  RSD.

REMARKS:

- % extracted - based on spike recovery.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN LANDFILL

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

---

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	69	64	2	0	2
s.w.		0.0121	0.0253	0.0000	
mean		0.0359	0.3078	0.0000	

---

QA CONTROL SAMPLES

---

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	204	1.731	0.1477	8.53

---

BLANKS

---

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	63	0.004	0.0044

---

ANALYTICAL PROCEDURE  
Inorganic Trace Contaminants Section

TEST NAME:Cobalt TEST CODE:COUT SAMPLE TYPE:Landfill Lch.  
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE: 533AA0  
REVISION NO: Original DATE:  
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 500 ml  
Container- Glass or plastic container  
Preservative- do not preserve  
Other- Field filtered samples preferred

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-93%  
Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS:mg/L to 3 places after decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.02 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 - 1.0 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls- A B  
mean 1.78 mg/L  
std. dev. 0.10 mg/L  
R.S.D. 5.6 %

Precision of Duplicates-low range mid range high range  
s.d. 0.007 0.007  
mean 0.035 0.405  
W 0.02 mg/L T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference QCLF1 exceed ±15% RSD.

REMARKS:

- % extracted - based on spike recovery.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT

IN LANDFILL

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20	to 0.50	0.50	to 1.0	>1.0
no.	111	24		1		0	1
s.w.		0.0067		0.0071		0.0000	
mean		0.0349		0.4050		0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	204	1.775	0.0994	5.60

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	47	0.002	0.0033

**ANALYTICAL PROCEDURE**  
**Inorganic Trace Contaminants Section**

TEST NAME:Copper            TEST CODE:CUUT            SAMPLE TYPE:Landfill Lch.  
UNIT:Ind.,Dom.,Landfill Wastes            SUPERVISOR:J. Pimenta

METHOD CODE: 533AA0

REVISION NO: Original

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 500 ml

Container- Glass or plastic container

Preservative- do not preserve

Other- Field filtered samples preferred

SAMPLE PREPARATION:Partial Extn.-      Total Extn.-Yes % Extracted-90%

Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS:mg/L to 3 places after decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.006 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.005 - 1.0 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

A

B

mean        1.84 mg/L

std. dev. 0.11 mg/L

R.S.D.        5.9 %

Precision of Duplicates-low range            mid range            high range

	s.d.	0.004	0.006	0.022
--	------	-------	-------	-------

W .005 mg/L	mean	0.040	0.369	0.616
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T .025 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference QCLF1 exceed ±15% RSD.

REMARKS:

- % extracted - based on spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.

- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER

IN LANDFILL

Operating Range = 0.005 to 1.0 mg/L

IN - RUN DUPLICATES

---

Range	<0.005	0.005 to 0.20	0.20	to 0.50	0.50	to 1.0	>1.0
no.	58	66		3		5	5
s.w.		0.0044		0.0059		0.0220	
mean		0.0401		0.3694		0.6161	

---

QA CONTROL SAMPLES

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SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	204	1.838	0.1092	5.94

---

BLANKS

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BLANK I.D.	NO.	MEAN	STD. DEV.
rb	76	0.006	0.0094

---

**ANALYTICAL PROCEDURE**  
Inorganic Trace Contaminants Section

TEST NAME:Iron                    TEST CODE:FEUT                    SAMPLE TYPE:Landfill Lch.  
UNIT:Ind., Dom., Landfill Wastes                    SUPERVISOR:J. Pimenta

METHOD CODE: 533AA0  
REVISION NO: Original                    DATE:  
NATURE OF LAST REVISION:

**SAMPLE HANDLING:**

Quantity Required- 500 ml  
Container- Glass or plastic container  
Preservative- do not preserve  
Other- Field filtered samples preferred

**SAMPLE PREPARATION:** Partial Extn.- Total Extn.-Yes % Extracted-92%  
Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

**INTERFERENCES:** Several, compensated for by the computer program.

**REPORTING RESULTS:** mg/L to 3 places after decimal.

**INSTRUMENTATION:** Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 50 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.02 mg/L

**PERFORMANCE CHARACTERISTICS:**

Routine Operating Range- 0.03 - 20.0 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

	A	B
mean	2.37 mg/L	
std. dev.	0.26 mg/L	
R.S.D.	11.0 %	
Precision of Duplicates-low range	mid range	high range
s.d.	0.11	0.32
mean	0.60	15.04
W 0.1 mg/L	T 0.5 mg/L	

**CONTROL LIMITS:** Analysis repeated if values for in-house reference QCLF1 exceed  $\pm 15\%$  RSD.

**REMARKS:**

- % extracted - based on spike recovery.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON IN LANDFILL

Operating Range = 0.030 to 20.0 mg/L

IN - RUN DUPLICATES

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Range	<0.030	0.030 to 4.00	4.00 to 10.00	10.00 to 20.0	>20.0
no.	18	93	11	5	10
s.w.		0.1078	0.0510	0.3223	
mean		0.6019	5.9441	15.0433	

---

QA CONTROL SAMPLES

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SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	197	2.370	0.2601	10.97

---

BLANKS

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BLANK I.D.	NO.	MEAN	STD. DEV.
rb	93	0.041	0.0516

---

**ANALYTICAL PROCEDURE**  
**Inorganic Trace Contaminants Section**

TEST NAME:Lead TEST CODE:PBUT SAMPLE TYPE:Landfill Lch.  
UNIT:Ind., Dom., Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE: 533AAO  
REVISION NO: Original DATE:  
NATURE OF LAST REVISION:

## SAMPLE HANDLING:

Quantity Required- 500 ml  
Container- Glass or plastic container  
Preservative- do not preserve  
Other- Field filtered samples preferred

**SAMPLE PREPARATION:** Partial Extn.- Total Extn.-Yes % Extracted-91%  
Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 3 places after decimal.

**INSTRUMENTATION:** Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 10 mg/L

**Resolution:**

Sensitivity:

Instrument Detection Limit: 0.02 mg/L

## **PERFORMANCE CHARACTERISTICS:**

Routine Operating Range- 0.03 - 1.0 mg/L

**Accuracy-** Not known; no reference standards available

Precision of Controls- A B

mean .738 mg/L

std. dev. .086 mg/L

R.S.D. 11.7 %

Precision of Duplicates-low range mid range high range

s.d. 0.047 0.115

**mean** 0.078 0.594

T 0.25 mg/L

CONTROL LIMITS: Analysis repeated if values for the house reference QCLF1 exceed  $\pm 15\%$  RSD.

**REMARKS.**

- % extracted - based on spike recovery.
  - Detection Limit - 3x std. dev. of low range within-run duplicates.
  - Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD

IN LANDFILL

Operating Range = 0.030 to 1.0 mg/L

IN - RUN DUPLICATES

---

Range	<0.030	0.030 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	121	11	0	2	3
s.w.		0.0472	0.0000	0.1149	
mean		0.0784	0.0000	0.5941	

---

QA CONTROL SAMPLES

---

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	201	0.738	0.0861	11.67

---

BLANKS

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BLANK I.D.	NO.	MEAN	STD. DEV.
rb	49	0.025	0.0329

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ANALYTICAL PROCEDURE  
Inorganic Trace Contaminants Section

TEST NAME: Magnesium      TEST CODE: MGUT      SAMPLE TYPE: Landfill Lch.  
UNIT: Ind., Dom., Landfill Wastes      SUPERVISOR: J. Pimenta

METHOD CODE: 533AAO  
REVISION NO: Original      DATE:  
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 500 ml  
Container- Glass or plastic container  
Preservative- do not preserve  
Other- Field filtered samples preferred

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-100%  
Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 3 places after decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 50 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.03 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.2 - 100 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

	A	B
mean	288.0 mg/L	
std. dev.	21.1 mg/L	
R.S.D.	7.3 %	

	low range	mid range	high range
s.d.	0.30	0.66	4.00
mean	8.65	27.7	71.61
W	0.5 mg/L	T 2.5 mg/L	

CONTROL LIMITS: Analysis repeated if values for in-house reference QCLF1 exceed  $\pm 15\%$  RSD.

REMARKS:

- % extracted - assumed 100% as material in solution.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%) .

SUMMARY REPORT OF QUALITY CONTROL DATA

MAGNESIUM IN LANDFILL

Operating Range = 0.200 to 100.0 mg/L

IN - RUN DUPLICATES

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Range	<0.200	0.200 to 20.00	20.00 to 50.00	50.00 to 100.0	>100.0
no.	6	64	25	21	21
s.w.		0.2951	0.6560	3.9991	
mean		8.6474	27.7172	71.6059	

---

QA CONTROL SAMPLES

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SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	204	288.016	21.1416	7.34

---

BLANKS

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BLANK I.D.	NO.	MEAN	STD. DEV.
rb	29	0.152	0.1474

---

**ANALYTICAL PROCEDURE**  
**Inorganic Trace Contaminants Section**

TEST NAME: Manganese      TEST CODE: MNUT      SAMPLE TYPE: Landfill Lch.  
UNIT: Ind., Dom., Landfill Wastes      SUPERVISOR: J. Pimenta

METHOD CODE: 533AAO  
REVISION NO: Original      DATE:  
NATURE OF LAST REVISION:

**SAMPLE HANDLING:**

Quantity Required- 500 ml  
Container- Glass or plastic container  
Preservative- do not preserve  
Other- Field filtered samples preferred

**SAMPLE PREPARATION:** Partial Extn.- Total Extn.-Yes % Extracted-94%  
Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

**INTERFERENCES:** Several, compensated for by the computer program.

**REPORTING RESULTS:** mg/L to 3 places after decimal.

**INSTRUMENTATION:** Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.003 mg/L

**PERFORMANCE CHARACTERISTICS:**

Routine Operating Range- 0.01 - 2.0 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

	A	B
mean	2.18 mg/L	
std. dev.	0.15 mg/L	
R.S.D.	6.6 %	
Precision of Duplicates-low range	mid range	high range
s.d.	0.005	0.024
mean	0.112	0.713
W 0.01 mg/L	T 0.05 mg/L	1.366

**CONTROL LIMITS:** Analysis repeated if values for in-house reference QCLF1 exceed ±15% RSD.

**REMARKS:**

- % extracted - based on spike recovery.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN LANDFILL

Operating Range = 0.010 to 2.0 mg/L

IN - RUN DUPLICATES

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Range	<0.010	0.010 to 0.40	0.40 to 1.00	1.00 to 2.0	>2.0
no.	10	88	15	10	14
s.w.		0.0049	0.0240	0.0327	
mean		0.1115	0.7130	1.3657	

---

QA CONTROL SAMPLES

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SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	204	2.181	0.1447	6.64

---

BLANKS

---

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	48	0.004	0.0028

---

ANALYTICAL PROCEDURE  
Inorganic Trace Contaminants Section

TEST NAME: Molydenum      TEST CODE: MOUT      SAMPLE TYPE: Landfill Lch.  
UNIT: Ind., Dom., Landfill Wastes      SUPERVISOR: J. Pimenta

METHOD CODE: 533AA0

REVISION NO: Original

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 500 ml

Container- Glass or plastic container

Preservative- do not preserve

Other- Field filtered samples preferred

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-100%  
Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 3 places after decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.005 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 - 1.0 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

A

B

mean .677 mg/L

std. dev. .108 mg/L

R.S.D. 15.9 %

Precision of Duplicates-low range mid range high range

s.d. 0.012

mean 0.060

W 0.01 mg/L

T 0.05 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference QCLF3 exceed  $\pm 15\%$  RSD.

REMARKS:

- Extraction efficiency assumed to be 100% as material originally in solution. No ref. stds. available.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MOLYBDENUM IN LANDFILL

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

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Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	123	13	0	1	0
s.w.		0.0120	0.0000	0.0243	
mean		0.0600	0.0000	0.5863	

---

QA CONTROL SAMPLES

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SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	204	0.677	0.1077	15.91

---

BLANKS

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BLANK I.D.	NO.	MEAN	STD. DEV.
rb	87	0.012	0.0109

---

**ANALYTICAL PROCEDURE**  
**Inorganic Trace Contaminants Section**

TEST NAME:Nickel            TEST CODE:NIUT            SAMPLE TYPE:Landfill Lch.  
UNIT:Ind.,Dom.,Landfill Wastes        SUPERVISOR:J. Pimenta

METHOD CODE: 533AAO  
REVISION NO: Original            DATE:  
NATURE OF LAST REVISION:

**SAMPLE HANDLING:**

Quantity Required- 500 ml  
Container- Glass or plastic container  
Preservative- do not preserve  
Other- Field filtered samples preferred

**SAMPLE PREPARATION:**Partial Extn.-      Total Extn.-Yes % Extracted-90%  
Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

**INTERFERENCES:** Several, compensated for by the computer program.

**REPORTING RESULTS:**mg/L to 3 places after decimal.

**INSTRUMENTATION:** Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.03 mg/L

**PERFORMANCE CHARACTERISTICS:**

Routine Operating Range- 0.01 - 1.0 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

	A	B
mean	1.75 mg/L	
std. dev.	0.15 mg/L	
R.S.D.	8.6 %	

Precision of Duplicates-low range	mid range	high range
s.d.	0.006	0.015
mean	0.034	0.279
W 0.05 mg/L	T 0.25 mg/L	0.012
		0.759

**CONTROL LIMITS:** Analysis repeated if values for in-house reference QCLF1 exceed  $\pm 15\%$  RSD.

**REMARKS:**

- % extracted - based on spike recovery.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

## SUMMARY REPORT OF QUALITY CONTROL DATA

## NICKEL IN LANDFILL

Operating Range = 0.010 to 1.0 mg/L

## IN - RUN DUPLICATES

Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	94	36	3	1	3
s.w.		0.0060	0.0148	0.0121	
mean		0.0338	0.2791	0.7587	

## QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	204	1.752	0.1503	8.58

## BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	80	0.005	0.0056

ANALYTICAL PROCEDURE  
Inorganic Trace Contaminants Section

TEST NAME:Vanadium      TEST CODE:VVUT      SAMPLE TYPE:Landfill Lch.  
UNIT:Ind.,Dom.,Landfill Wastes      SUPERVISOR:J. Pimenta

METHOD CODE:533AA0

REVISION NO: Original

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 500 ml

Container- Glass or plastic container

Preservative- do not preserve

Other- Field filtered samples preferred

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-100%

Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS:mg/L to 3 places after decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.008 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.010 - 1.0 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

A

B

mean .587 mg/L

std. dev. .156 mg/L

R.S.D. 26.5 %

Precision of Duplicates-low range mid range high range

s.d. 0.059 0.015

mean 0.050 0.261

W 0.02 mg/L T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference QCLF1 exceed  $\pm 15\%$  RSD.

REMARKS:

- % extracted - assumed 100% as material in solution.

- Detection Limit - 3x std. dev. of low range within-run duplicates.

- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM IN LANDFILL

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

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Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	114	22	1	0	0
s.w.		0.0593	0.0151	0.0000	
mean		0.0503	0.2613	0.0000	

---

QA CONTROL SAMPLES

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SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	184	0.587	0.1557	26.54

---

BLANKS

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BLANK I.D.	NO.	MEAN	STD. DEV.
rb	48	0.009	0.0066

---

**ANALYTICAL PROCEDURE**  
**Inorganic Trace Contaminants Section**

TEST NAME:SELENIUM TEST CODE:SEUT SAMPLE TYPE:Landfill Lch.  
UNIT: Biomaterials SUPERVISOR: R. Sadana

METHOD CODE:510BF3  
REVISION NO:  
NATURE OF LAST REVISION:

DATE: January, 1983

#### SAMPLE HANDLING:

**Quantity Required- 100 ml**

Container- Glass bottle with bakelite screw cap (16 oz.)

Preservative-1ml conc. HNO<sub>3</sub> for sample filling 16 oz. plastic bottle with Bakelite screw cap (16 oz.)

**Other-**

**SAMPLE PREPARATION:** Partial Extn.- Total Extn.-yes % Extracted-  
Procedure- Digest samples by mixing 20 ml of sample and 4ml of acid (6:3:1 HNO<sub>3</sub>:HClO<sub>4</sub>:H<sub>2</sub>SO<sub>4</sub>) and heating on a hot plate until dense white fumes are formed. Allow sample to cool and add .5 ml of H<sub>2</sub>O and 2.5 ml HCl. Transfer the contents of the beaker to a test tube graduated at the 20 ml mark and bring to volume with washings. Shake and analyze by automated hydride-F.A.A.S. technique.

**INTERFERENCES:** Excessive concentrations of Cu, Fe, Ni

**REPORTING RESULTS:** mg/L-2 dec. if  $\leq$  10, 1 dec. if  $10-100$ , 0 dec. if  $>100$

**INSTRUMENTATION:** Atomic Absorption Spectrophotometer (Varian 1200) with strip chart recorder, peristaltic pump, auto-sampler, open-ended and heated quartz 'T' cell ( $0.6 \times 10$  cm), and gas-liquid separator.

Calibration Range: 0 to 40  $\mu$ g/ml

Resolution: 0.01 absorbance

Sensitivity: 20  $\mu$ g/ml gives 0.200 Abs.

Instrument Detection Limit: 1 ng/ml

#### **PERFORMANCE CHARACTERISTICS:**

Routine Operating Range - 0.001 to 0.04 mg/l

### **Accuracy-**

### Precision of Controls-

A

mean .075 mg/L

std. dev. .004

8

R.S.D. 5.3 %

cates low range  
as  $\beta = 0.0002$

B.d. 0.000

W -001 mg/L

T = 0.05 mg/L

### high range

#### **CONTROL LIMITS.**

**REMARKS -**

SUMMARY REPORT OF QUALITY CONTROL DATA

SELENIUM IN LANDFILL

Operating Range = 0.001 to 0.04 mg/L

IN - RUN DUPLICATES

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Range	<0.001	0.001 to 0.01	0.01 to 0.02	0.02 to 0.04	>0.04
no.	24	9	0	0	2
s.w.		0.0002	0.0000	0.0000	
mean		0.0020	0.0000	0.0000	

---

QA CONTROL SAMPLES

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SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
475-3	34	0.075	0.0040	5.33

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BLANKS

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BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

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ANALYTICAL PROCEDURE  
Inorganic Trace Contaminants Section

TEST NAME: Zinc TEST CODE: ZNUT SAMPLE TYPE: Landfill Lch.  
UNIT: Ind., Dom., Landfill Wastes SUPERVISOR: J. Pimenta

METHOD CODE: 533AAO  
REVISION NO: Original DATE:  
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 500 ml  
Container- Glass or plastic container  
Preservative- do not preserve  
Other- Field filtered samples preferred

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-88%  
Procedure- Do not shake samples. Transfer 50 ml of sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml conc. HNO<sub>3</sub> and dry at 105°C in a forced air oven. Add 2.0 ml of aqua-regia to the test tube and digest for 2 hours. Make volume to 12.5 ml with distilled water and mix well. Determine the elemental concentration by ICP emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 3 places after decimal.

INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Jobin-Yvon JY48P, equipped with autosampler and DEC computer system for concentration print-out; Commodore Pet microcomputer interface to LIS.

Calibration Range: 0 - 10 mg/L

Resolution:

Sensitivity:

Instrument Detection Limit: 0.005 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 - 1.0 mg/L

Accuracy- Not known; no reference standards available

Precision of Controls-

A

B

mean 1.75 mg/L

std. dev. 0.18 mg/L

R.S.D. 10.1 %

Precision of Duplicates-low range mid range high range

s.d. 0.019 0.007 0.014

mean 0.045 0.272 0.920

W 0.02 mg/L T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference QCLF1 exceed  $\pm 15\%$  RSD.

REMARKS:

- % extracted - based on spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.  
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC

IN LANDFILL

Operating Range = 0.010 to 1.0 mg/L

IN - RUN DUPLICATES

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Range	<0.010	0.010 to 0.20	0.20 to 0.50	0.50 to 1.0	>1.0
no.	52	65	7	1	12
s.w.		0.0186	0.0068	0.0141	
mean		0.0450	0.2715	0.9200	

---

QA CONTROL SAMPLES

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SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCLF-3	201	1.748	0.1769	10.12

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BLANKS

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BLANK I.D.	NO.	MEAN	STD. DEV.
rb	103	0.023	0.0245

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**ANALYTICAL PROCEDURE**  
Inorganic Trace Contaminants Section

TEST NAME: Total cyanide TEST CODE: CCNAUR SAMPLE TYPE: Landfill Lch.  
UNIT: QC-Project SUPERVISOR: J. Hipfner

METHOD CODE: 001AC2

REVISION NO:

DATE

NATURE OF LAST REVISION:

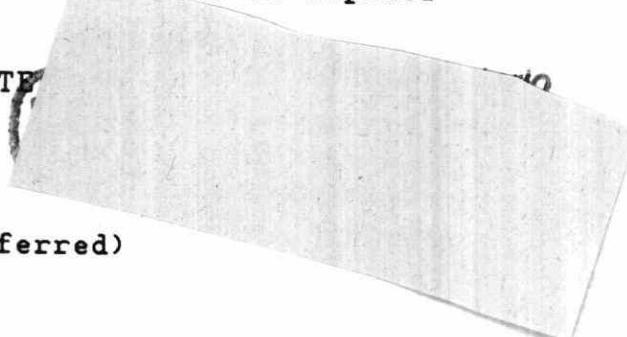
SAMPLE HANDLING:

Quantity Required- 500 ml

Container- Glass or plastic (preferred)

Preservative- NaOH

Other-



SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-100

Procedure- The sample is first run to see if there is cyanide present. This is run directly by the automated high temperature distillation with 25% H<sub>3</sub>PO<sub>4</sub>-5% H<sub>3</sub>PO<sub>2</sub> followed by a colourimetric analysis with chloramine T -isonicotinic acid -barbituric acid method.

If the total cyanide is > .01 mg/L then 5 to 250 ml of sample is manually distilled with 30 ml of 15%(w/v)tartaric acid. The distillate is collected in 50 ml of 1N NaOH, and analyzed by the automated Technicon distillation system referred to above.

INTERFERENCES: SCN interference is removed by distillation.

Distillable organics may interfere; also S= at high levels.

REPORTING RESULTS: Mg/l CN: 3 decimal places up to 3 significant figs

INSTRUMENTATION: Technicon AAI continuous flow analyzer including pump, colourimeter, appropriate autosampler and recorder. High temperature distillation apparatus (Technicon). Manual dist. app

Calibration Range: 0 to 0.2 mg/l as CN

Resolution: 0.001 mg/l

Sensitivity:

Instrument Detection Limit: 0.001 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.00100 to 0.200 mg/l

Accuracy- 100%

Precision of Controls-

	A	B
mean	.110 mg/L	0.059
std. dev.	.0027mg/L	0.0026
R.S.D.	2.45 %	4.41 %

Precision of Duplicates-low range mid range high range

s.d.

mean

W .001 mg/L

T .005 mg/L

CONTROL LIMITS:

REMARKS: Pure CN standards are recovered 100% during manual distillation. Complex cyanides can normally be expected to be recovered at 100%.

SUMMARY REPORT OF QUALITY CONTROL DATA

TOTAL CYANIDE IN LANDFILL

Operating Range = 0.001 to 0.4 mg/L

IN - RUN DUPLICATES

Range	<0.001	0.001 to 0.08	0.08 to 0.2	0.2 to 0.4	>0.4
no.	0	2	0	0	0
s.w.		0	0	0	
mean		0.011	0	0	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	146	0.149	0.0049	3.29
qc-b	146	0.018	0.0022	12.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	146	0.001	0

ANALYTICAL PROCEDURE  
Inorganic Trace Contaminants Section

TEST NAME: Free cyanide TEST CODE: CCNFUR SAMPLE TYPE: Landfill Lch.  
UNIT: QC-Project SUPERVISOR: J. Hipfner

METHOD CODE: 700AC2

REVISION NO:

DATE:

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Glass or plastic (preferred)

Preservative- NaOH

Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted- \*  
Procedure- Pass sample aliquot through an automated low temperature distillation (106°C) in a distillation acid consisting of 10% acetic acid and 0.5% zinc acetate.  
Analyze distillate by the Chloramine-T -pyridine-barbituric acid colourometric method, or equivalent.

INTERFERENCES: None

REPORTING RESULTS: Mg/l CN to 2 decimal places up to 3 significant figs

INSTRUMENTATION: Technicon automated continuous flow analyzer including pump, colourimetric distillation apparatus and sampler; suitable recorder.

Calibration Range: 0 to 0.2 mg/l as CN

Resolution: 0.001

Sensitivity:

Instrument Detection Limit: 0.001 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0100 to 0.400 mg/l

Accuracy- 100%

Precision of Controls-

	A	B
mean	.110 mg/L	0.060
std. dev.	.0036mg/L	0.0031
R.S.D.	3.27 %	5.17%

Precision of Duplicates-low range mid range high range

s.d.

mean

W .001 mg/L

T .005 mg/L

CONTROL LIMITS:

REMARKS: \* The test defines the results reported in this case. The terminology "Weak Acid Dissociable" is commonly used and represents weakly associated cyanide compounds such as KCN, NaCN, NiCN<sub>4</sub>, HCN, etc.

SUMMARY REPORT OF QUALITY CONTROL DATA

FREE CYANIDE IN LANDFILL

Operating Range = 0.001 to 0.4 mg/L

IN - RUN DUPLICATES

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Range	<0.001	0.001 to 0.08	0.08 to 0.2	0.2 to 0.4	>0.4
no.	0	2	0	0	0
s.w.	0	0	0	0	0
mean	0.001	0	0	0	0

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QA CONTROL SAMPLES

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SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	135	0.151	0.0062	4.11
qc-b	135	0.018	0.0022	12.22

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BLANKS

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BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	135	0.001	0

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